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(U) PERFORMANCE OF A PYROLYTIC GRAPHITE REINFORCED GRAPHITE FELT NOZZLE INSERT FIRED WITH A FLUORINATED PROPELLANT

AD509011

D. L. RIEDL, 1/LT, USAF

TECHNICAL REPORT AFRPL-TR-69-11

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AFRPL-TR-69-11

PERFORMANCE OF A PYROLYTIC GRAPHITE REINFORCED GRAPHITE FELT NOZZLE INSERT FIRED WITH A FLUORINATED PROPELLANT (U)

Don L. Riedl, 1Lt, USAF

January 1969

In addition to security requirements which must be met, this document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of AFRPL (RPOR/STINFO), Edwards, CA 93523.

FOREWORD

(U) This report covers the design and test evaluation accomplished on a nozzle insert developed under an Air Force Rocket Propulsion Laboratory (AFRPL) contract. This contract, F04611-67-C-0060, with Philco-Ford Corp was under the cognizance of Robert Hale, the Philco Project Engineer. Air Force direction was provided by 1st Lt D. L. Riedl, RPRRE, Engine Research Branch. The testing described by this report occurred during July and August 1968 at the AFRPL under Project Flintstone (305803FRC) which was directed by Mr. Gail Bergen.

(U) This report contains no classified information extracted from other classified documents.

(U) This report has been reviewed and is approved.

HOWARD V. MAIN, GS-14
Chief of In-House Liquid Rocket Division
Air Force Rocket Propulsion Laboratory

CONFIDENTIAL ABSTRACT

(U) This report presents the results of testing performed on a nozzle insert which utilized pyrolytic graphite reinforced graphite felt as the throat material. The background for this testing, the design description, and the test conditions are also reported.

(U) Three tests were made to complete the 300 sec duty cycle. The nominal thrust was 3000 lbs, and chamber pressure was 300 psia. The mixture ratio was 2.85, which is the optimum value for C* performance for the chlorine trifluoride/monomethylhydrazine (CTF/MMH) propellant combination used in this testing. The duty cycle included hot and cold restarts.

(C) The erosion resistance of the throat material was found to be good, as no measurable erosion was experienced. There was a significant effect of the MMH fuel with regard to the carbon deposition phenomena in the nozzle throat. Structurally, the insert performed well, and the thermal insulation material proved adequate.

(U) The results of this testing conclusively support that graphite materials are excellently suited to withstand the environment of the CTF/MMH propellant combination.

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SECTION I

INTRODUCTION

(U) The nozzle insert described in this report was developed and tested under Contract F04611-67-C-0060 with the Philco-Ford Corporation. This contract was generated by the introduction of fluorinated propellants to the rocket industry. Fluorinated rocket exhausts are very severe, both chemically and thermally, and therefore new materials were required before fluorine oxidizers could be used in rocketry. Prior to this contract, material testing had been done using fluorinated propellants. However, these investigations were done with several different materials used in different configurations and at various chamber pressures, as well as for varying duty cycles. Contract F04611-67-C-0060 was designed to pick the materials which had demonstrated good performance under these previous programs, and evaluate them under similar design and test conditions. The specific objective of this contract was to establish properties of candidate materials, pick the three best materials based on their properties, and test these three materials in the form of a nozzle insert using liquid fluorine (LF_2), chlorine trifluoride (CTF) oxidizer with a hydrazine blend fuel. A total of six inserts were provided. This report will be concerned with describing the performance of the first of the two nozzle inserts which used pyrolytic graphite reinforced felt (RPG) as the throat material. The propellant combination was CTF/MMH, which has a 5700°F theoretical combustion temperature at the optimum mixture ratio used in this testing.

SECTION II

TEST ARTICLE DESCRIPTION

A. DESIGN CONDITIONS

(U) Under Contract F04611-67-C-0060, criteria were specified to which the final insert assemblies were to be designed. For the insert being described in this report, the following design criteria were used:

Propellants	CTF/MMH
Pc	300 psia (exhaust to 13.2 psia)
Thrust	3000 lbs (S. L.)
Mixture Ratio	2.4 - 2.8
Performance	92 Percent Isp
Duty Cycle	300 sec (30 restarts)

B. MATERIALS USED

(U) The final insert design using RPG as the throat insert material is shown in Figure 1. Each section of the design, identified by circled numerals will be described below.

(U) 1 This is the RPG insert which was of principal interest in the testing. Its function was to remain structurally sound throughout the duty cycle while maintaining a constant or very slowly changing throat diameter. The RPG was to be evaluated for its ability to withstand thermal shock, to resist erosion and chemical corrosion, and to remain intact structurally while accommodating large thermal gradients. RPG, which is graphite felt impregnated with pyrolytic graphite, is a product of Super Temp Corporation. The raw material used in making RPG is a standard rayon felt. This felt is placed in a furnace and carbonized at about 1550°F by hydrocarbon cracking of the rayon molecules. The carbon skeleton is then raised to 5000°F which induces the carbon atoms to orient themselves in a graphite crystal structure. At this point,

graphitization of the rayon felt is complete. Impregnation of the graphite felt with pyrolytic graphite is the next step toward the finished product. In this process the felt substrate is placed in a furnace which is raised to about 5400°F. At this temperature, methane gas is introduced. The methane is dehydrogenated as it passes through the graphite fibers of the felt and the carbon is deposited on the fibers in the form of pyrolytic graphite. This impregnation process is continued until the desired density is reached. As a general rule, the highest density possible is desired, since higher density materials have demonstrated better resistance to erosion in past testing. The density of the RPG insert described by this report was 1.62 g/cm^3. As a result of its random orientation of pyrolytic graphite, RPG is a more thermally isotropic material than virgin pyrolytic graphite, since the highly directional thermal conductivity of pyrolytic graphite is averaged by the random P.G. orientation. The result is that the effective thermal conductivity of RPG is lower than pyrolytic graphite wafers, and therefore the surface temperature of RPG will rise faster. The significance of the fast rising surface temperature is that it lowers the heat flux into the backside material, allowing for longer firing times with a given amount of insulation.

(U) (2) Material in the entrance section in this position was ATJ graphite. Its function was to reduce erosion in the area where the gases were converging to the throat.

(U) (3) In the expansion region, the material used was Speer 8882 graphite. This is a bulk graphite almost identical to ATJ graphite. The function of this section was to prevent serious erosion in the divergent section of the insert.

(U) (4) The material directly behind the insert material was Grade GA hard carbon. Its function was to act as an intermediate insulator which was structurally compatible with the insert material. GA hard carbon was chosen for this use because it was a good insulator with high

temperature capability. Also, GA hard carbon can be repeatedly heated to high temperatures without experiencing significant property changes.

(U) 5 This graphite cloth phenolic washer section was placed between the ATJ graphite section and the water-cooled thrust chamber to which the insert was attached. The reason for placing this graphite phenolic washer here was to lower the thermal gradient from the ATJ graphite surface to the water-cooled chamber. Without this section of graphite phenolic, the temperature difference between the ATJ graphite and the chamber would have been about $4000^{o}F$. The heat flux into a localized area of the chamber due to this high thermal gradient would be enough to melt the metal and cause the chamber to fail. However, the graphite phenolic washer lowers the temperature differential to about $1000^{o}F$ at the washer-chamber interface, and thus eliminates the possibility of destructive heat flux into the water-cooled chamber. This washer was replaced between each firing because it was pyrolized during the firings and therefore structurally incapable of cold restarts. Since the washer was not being evaluated, this replacement procedure did not detract from the results of testing.

(U) 6 and 7 The material used in both of these regions was a low grade asbestos phenolic made by pressing strands of chopped asbestos phenolic rope together. The function of the asbestos phenolic was to insulate the steel holder from the high temperature carbon. Asbestos phenolic was used because it is a good low temperature insulator.

(U) 8 and 9 347 stainless steel and 1020 carbon steel were used as the insert holder and aft closure plate, respectively. Both were workhorse designs, and were provided by the Air Force Rocket Propulsion Laboratory (AFRPL).

SECTION III

TEST HARDWARE AND CONDITIONS

A. INJECTOR

(C) For the testing of this insert an existing concentric ring like-doublet injector was used. This injector had originally been designed for use with an LF_2 oxidizer, but was modified for use with CTF. Two major changes were made to the injector. The orifice pattern was reduced from an 8 in. diameter to a 6 in. diameter by simply closing the outer orifices. Also, on the resulting outside oxidizer doublet ring, the outer orifices were all increased in diameter to tip the resultant oxidizer stream inward, thus lessening the change of injector streaking. These changes also provided a mild degree of zone cooling. There are 289 doublet elements consisting of 144 fuel and 145 oxidizer elements. The injector face is made of nickel and the flange is made of 347 stainless steel. During the injector check-out phase of the testing, the injector proved to be stable and high performing (92-93 percent Isp), as well as durable. Figure 2 shows this injector prior to firing of the insert.

B. THRUST CHAMBER

(U) To test the inserts it was necessary to provide a thrust chamber. For the testing of this insert, a 10 in. long, 6 in. I.D. water-cooled workhorse chamber was used for durability and for smoothing of injector streaking in the gaseous wall environment before the gases reached the inserts. The chamber had a "ROCKIDE" coating which served to decrease heat flux into the walls and to prevent exhaust erosion of the chamber.

C. TEST STAND

(U) Firings on this insert were conducted at the 1-46 test area using pad A-2. For the insert evaluation, venturies were used in both fuel and oxidizer feed lines. The tank pressures used to assure cavitation in both

venturies were 1500 psig in the MMH vessel and 1700 psig in the CTF vessel. Storage capacity of the tanks was sufficient (250 gal MMH, 500 gal CTF) to allow durations in excess of 200 sec for the conditions at which this insert was to be fired.

D. INSTRUMENTATION

(U) Analog and digital recording of water tank, propellant tank, chamber, and injector manifold pressures, thrust and three temperature readings were used for this testing. In addition, digital recording of water and propellant flowrates, and nine more temperatures were used. For this insert a total of eleven temperature recording channels were used, two on analog and nine on digital read out. Specific locations of the eleven chromel-alumel thermocouples used for this insert are given in Table I.

E. TEST CONDITIONS

(U) Test conditions which were set for the RPG insert were the same as the design conditions. These included:

300 psia	Pc
3000 lbs	Thrust
2.85	Mixture Ratio

The specific duty cycle formulated was as follows:

First Firing	Second Firing	Third Firing
100 sec on	50 sec on	20 pulses of 5 sec on, 5 sec off
cool to ambient	5 sec off 10 pulses of 5 sec on, 5 sec off cool to ambient	cool to ambient

This duty cycle would allow a good thermal evaluation of the materials and design to be made, plus demonstrating the cold and hot restart capability of the material. A very thorough evaluation of this insert could therefore be made by using this duty cycle.

SECTION IV

TEST RESULTS

A. ACTUAL DUTY CYCLE

(C) The actual duty cycle to which this insert was subjected is listed below:

First	Second	Third
100 sec cool to ambient	50 sec on, 5 sec off 10 pulses of 5 sec on, 5 sec off cool to ambient	17 pulses of 5 sec on, 5 sec off cool to ambient abort

(C) At the termination of the first firing, the Pc had risen from 300 psia to 555 psia. This Pc rise occurred as a slow, gradual rise throughout the run. After examining the test hardware at the conclusion of the firing it was observed that there had been a carbon buildup in the insert causing an effective reduction in the throat area, and thus the Pc rise. The source of carbon in this instance was the methyl (CH_3) group which is contained in the fuel monomethylhydrazine. This fuel releases the carbon on combustion. Ultimately this carbon will be either deposited on the chamber wall or accelerated out of the throat with the rest of the exhaust gases. In this instance, a substantial amount of carbon was deposited on the wall of the chamber. Figure 3 illustrates the manner in which the carbon was deposited.

(U) Examination of the injector following this first test revealed that it had sustained rather severe face burning. This necessitated replacing the injector with one of the same design which was being held

in reserve for such a mishap. The only reason which can be given to explain what caused the injector face burning is the rise in Pc. It is believed the higher heat flux into the injector face at the high Pc condition was sufficient to exceed the cooling capability of the propellant manifolding and thus cause the face burning. The burned injector is shown in Figure 4.

(C) The second firing was accomplished as planned. The fuel flow was high and therefore the actual mixture ratio of 2.4 was below the 2.85 desired. However, the Pc was 315 psia and the thrust just over 3000 lbs. So, in spite of the low mixture ratio the test was considered a success because the capability of hot restarts was demonstrated at conditions very near those desired.

(C) Termination of the third test was premature due to a very erratic chamber pressure trace. The test was intended to consist of twenty 5 sec pulses each followed by a 5 sec off time. On the fifteenth pulse of this firing the Pc began to fluctuate quite sharply, and on the seventeenth pulse it was decided to terminate, even though this pressure fluctuation was not shown on the propellant manifold pressure traces. Investigation following the test revealed that the Pc port was obstructed with carbon released by the MMH on combustion. In spite of the abort, the test was considered successful, as cold restart and cold pulsing capability were demonstrated.

B. POST TEST MATERIALS ANALYSIS

(C) The post test analysis gave a good indication of the material performance, and therefore a brief description of the condition of each of the insert materials after this test series will be given. The circled numbers again correlate to those in Figure 1.

(C) 1 This RPG material survived the 285 sec firing in excellent condition. No change occurred in the entrance or exit angles, nor in the

length of the insert. The original throat dimension was also unchanged. The only erosion which occurred was found in three isolated spots on the exit section of the insert. The depth of this erosion was measured to be 0.05 in. There was also a coating of pyrolytic graphite deposited on the insert. This P.G. coating was formed from the carbon in the exhaust released by the MMH fuel. It was found that the thickness of the P.G. decreased from the entrance to the exit. An explanation of the deposition of the P.G. is that the RPG has many very good growth sites from which P.G. crystals can begin their formation. These growth sites coupled with the high surface temperature of the RPG during firing create a condition which is ideally suited to P.G. deposition. The RPG was not sectioned because it could be fired again and contained no material internally which could be altered by heat. The insert is shown in Figure 5 with the carbon and P.G. removed.

(C) 2 The ATJ graphite entrance section was found to be cracked in three places in the axial direction. The cracks were located at 0°, 110°, and 200° when referenced to the firing position of the part, 0° being 12 o'clock. These cracks were expected, based on performance results of this material in previous firings on other programs. This material was also coated with pyrolytic graphite. Figure 6 shows this piece.

(C) 3 The expansion section material, Speer 8882 graphite, contained two small axial cracks, neither of which extended completely through the part. Some pyrolytic graphite was also found on the Speer 8882 and minor erosion of the surface was observed.

(C) 4 The GA carbon high temperature insulation section was cracked through in four places. Their locations were 0°, 90°, 180° and 255° referenced to the firing position, 0° being 12 o'clock. These cracks were expected to occur. Figure 7 shows this part.

(C) (5) This graphite phenolic washer was replaced during each firing sequence and was not evaluated.

(C) (6) This backside insulator was charred to a depth of about 3/8 in. The material had some minor cracks but overall was in very good condition. Figure 8 exhibits this material.

(C) (7) The aft insulator was charred about 3/8 in. from the Speer graphite surface, and it had been compressed about .07 in. by the axial expansion of the insert during firing. A portion of the exposed surface was removed by plume impingment. This no doubt occurred during the first 100 sec firing when the Pc rise was experienced. The nozzle was designed for a plume resulting from a 300 psia chamber pressure, and the plume at 350 psia was large enough to significantly impinge upon the asbestos insulator.

(C) (8) and (9) Neither of these steel holding members were significantly affected by the testing. Section (9) did have some material removed during the 100 sec firing, as is described for the material in Section (7) above.

(C) Figure 9 provides a good summarizing view of the post test findings on the RPG insert. Figure 10 provides an illustration of the post test insert.

(C) Figure 11 shows the thermal response of the insert during the 100 sec firing. These temperature responses were low and did not rise as rapidly as expected. The reason for the low readings is suspected to be improper seating of the thermocouples in the materials whose temperatures were to be measured. Although no accurate value of the wall temperature during this run can be determined, the presence of P.G. deposition on the insert indicates that temperatures in the vicinity of

3500 to 4000°R were attained. This temperature is not hot enough to induce severe hydrogen fluoride (HF) attack on the carbon wall, which helps explain the fact that no throat erosion was experienced during the firing of this insert.

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

(C) The general conclusion which has been drawn from the test and post-test results is that the insert design was sound and that RPG is a good material candidate for use with fluorinated propellants. The first firing of 100 sec demonstrated the thermal response and erosion resistance of the RPG and design as a whole. The second firing demonstrated cold and hot restart capability of the material and design configuration. An ability to withstand pulse firing when cold was exhibited by the insert in the third firing series. These demonstrated qualities of the insert materials and design suggest that they are quite durable and could be used in a wide range of applications with a fluorinated oxidizer at the 3K thrust and 300 psia Pc range of operation.

(C) The erosion results of this testing, as promising as they are, must be tempered by the fact that MMH was used as the fuel. As mentioned before, combustion of CTF and MMH releases carbon. This carbon saturates the exhaust gases as free carbon at equilibrium. As a result, the amount of corrosive HF in the exhaust is reduced before it reaches the wall of the chamber, making the chemical attack much weaker than would be experienced when using other hydrazine fuels. In fact, there is a carbon buildup on the chamber and nozzle wall.

(U) Recommendations for further evaluation of RPG would be to use a different fuel. Hydrazine fuels which would eliminate free carbon from the exhaust and thus provide a more severe chemical environment in which to evaluate the RPG should be used.

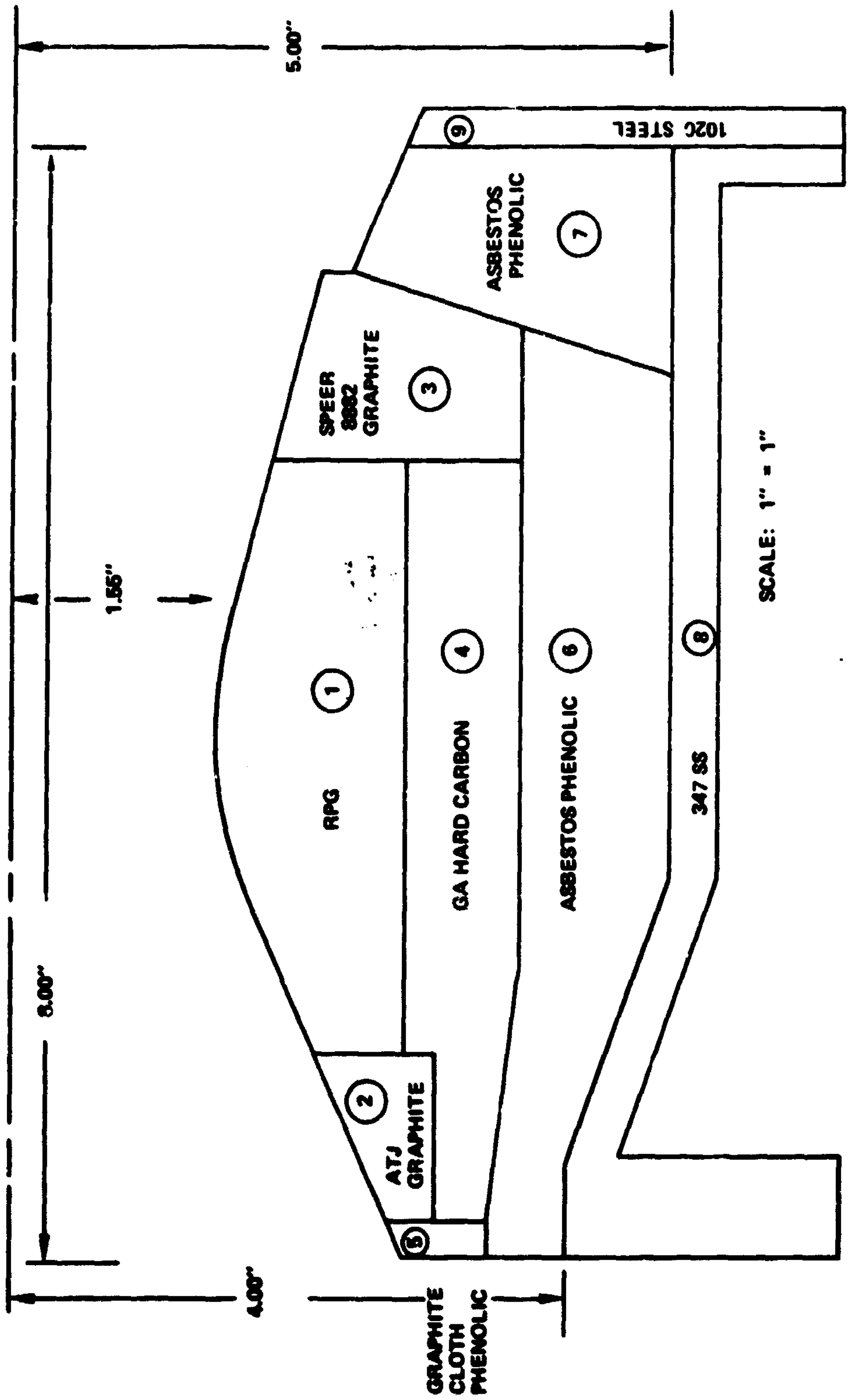

Figure 1. Philco CTF Insert

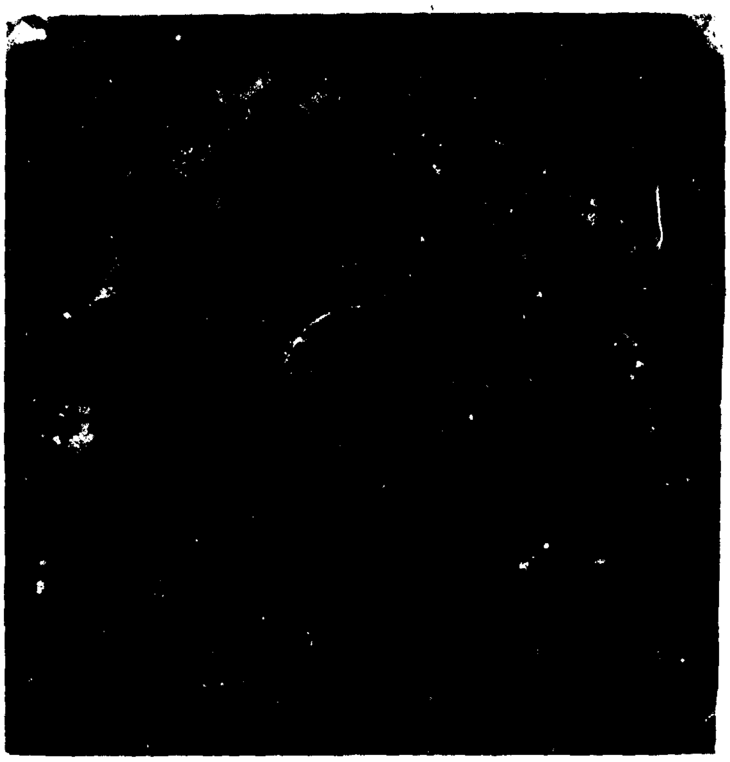

Figure 2. Injector No. 25-N2

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Figure 3. Post Fire Carbon Deposition

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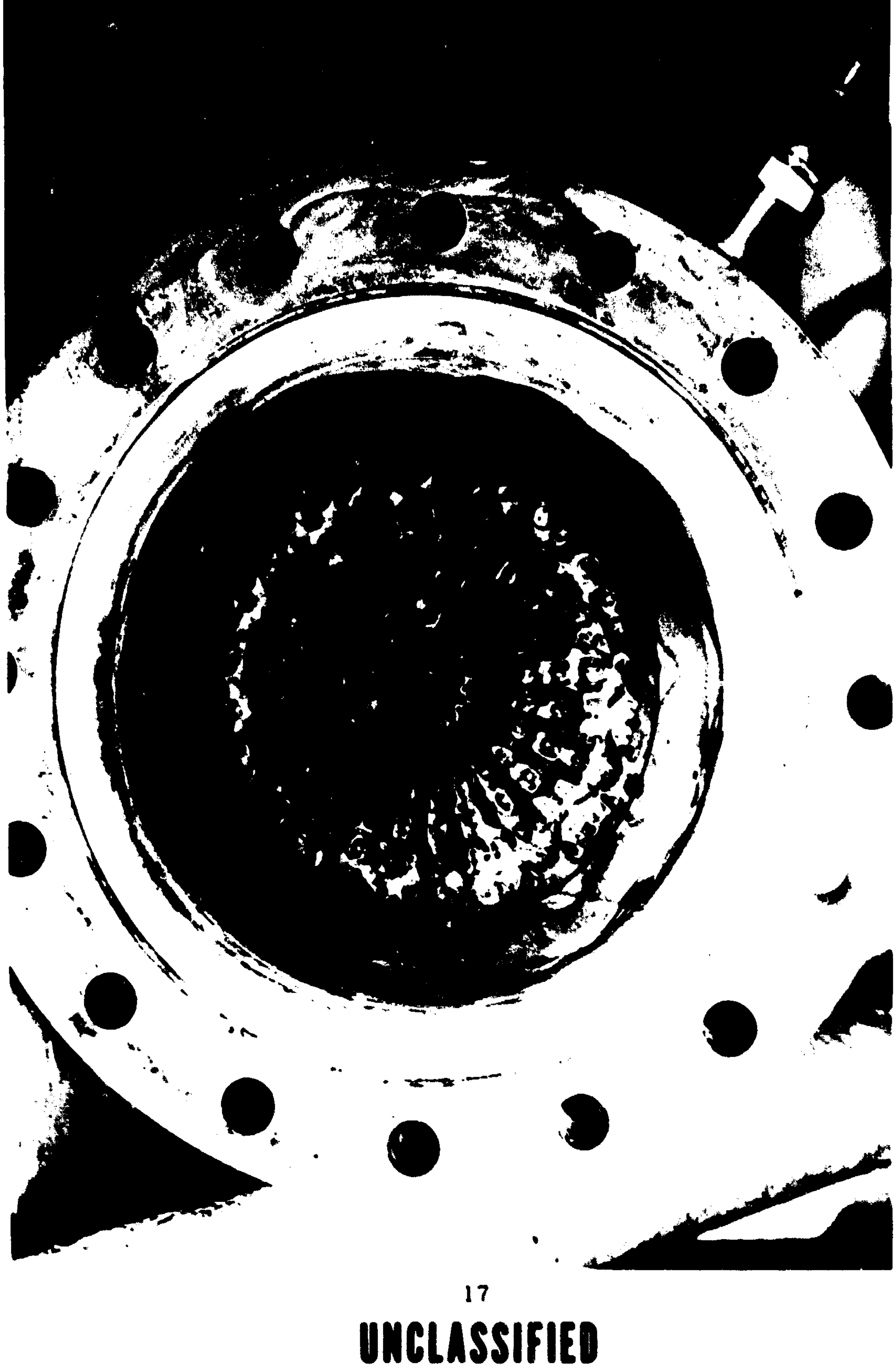


Figure 4. Damaged Injector No. 25-N2

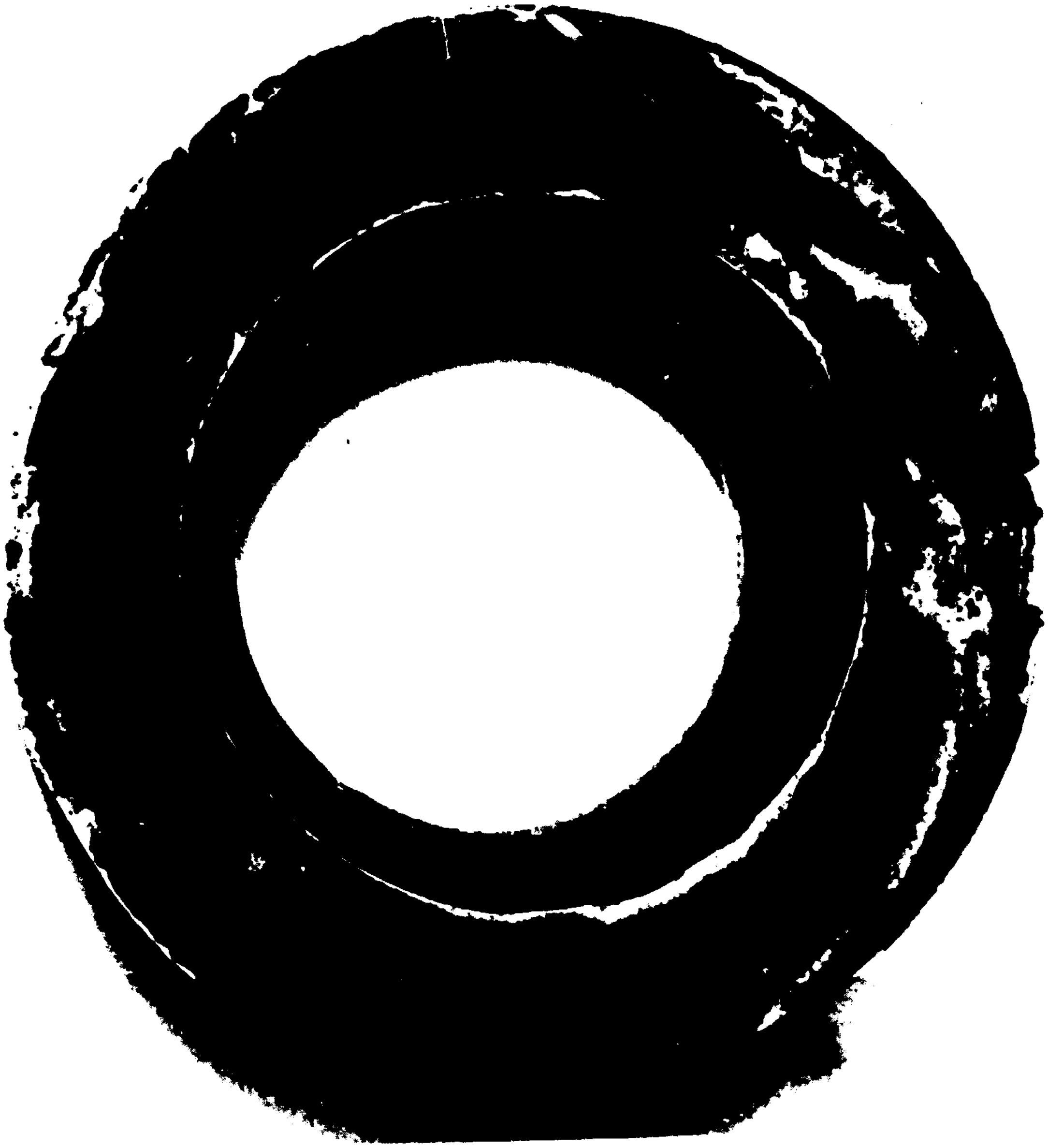


Figure 5. Post Test RPG Insert Section

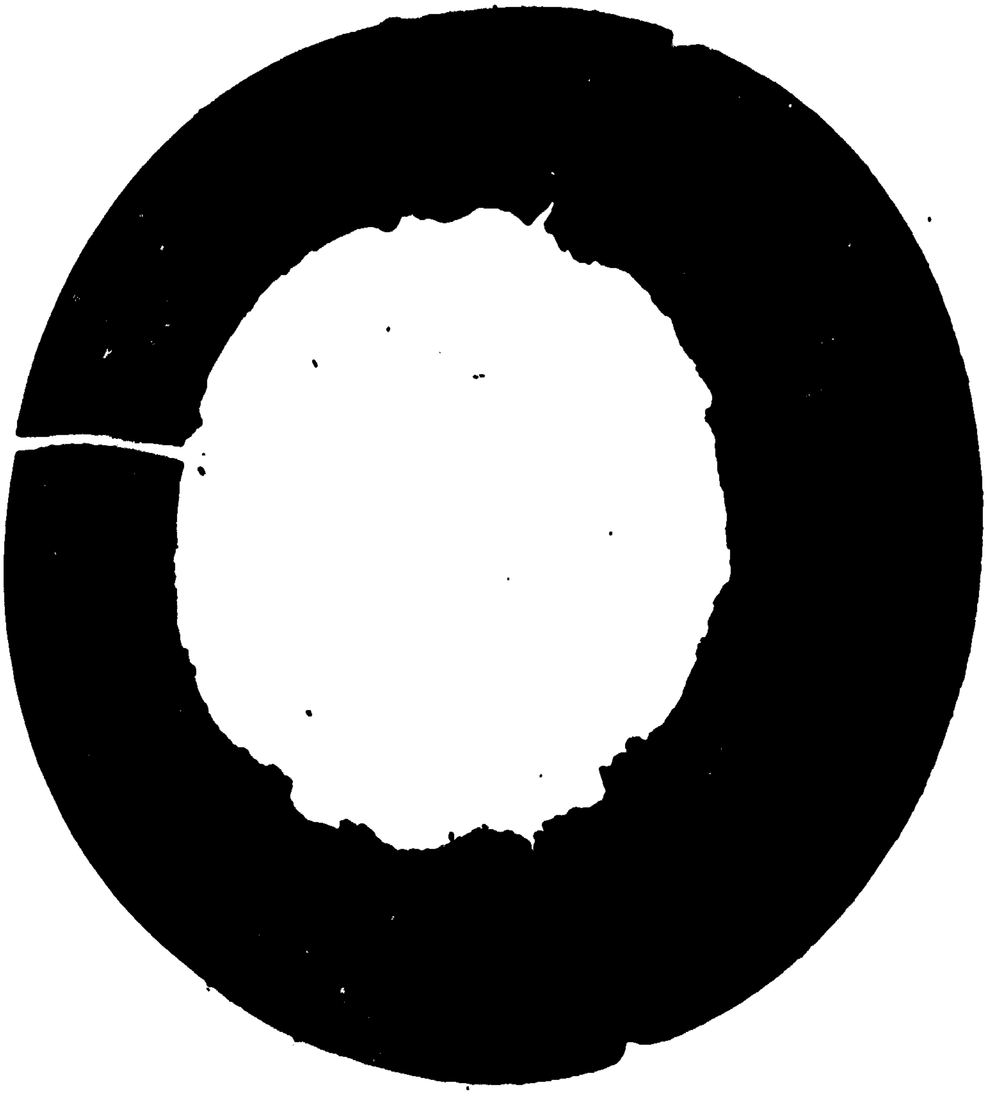


Figure 6. Post Test ATJ Graphite Section

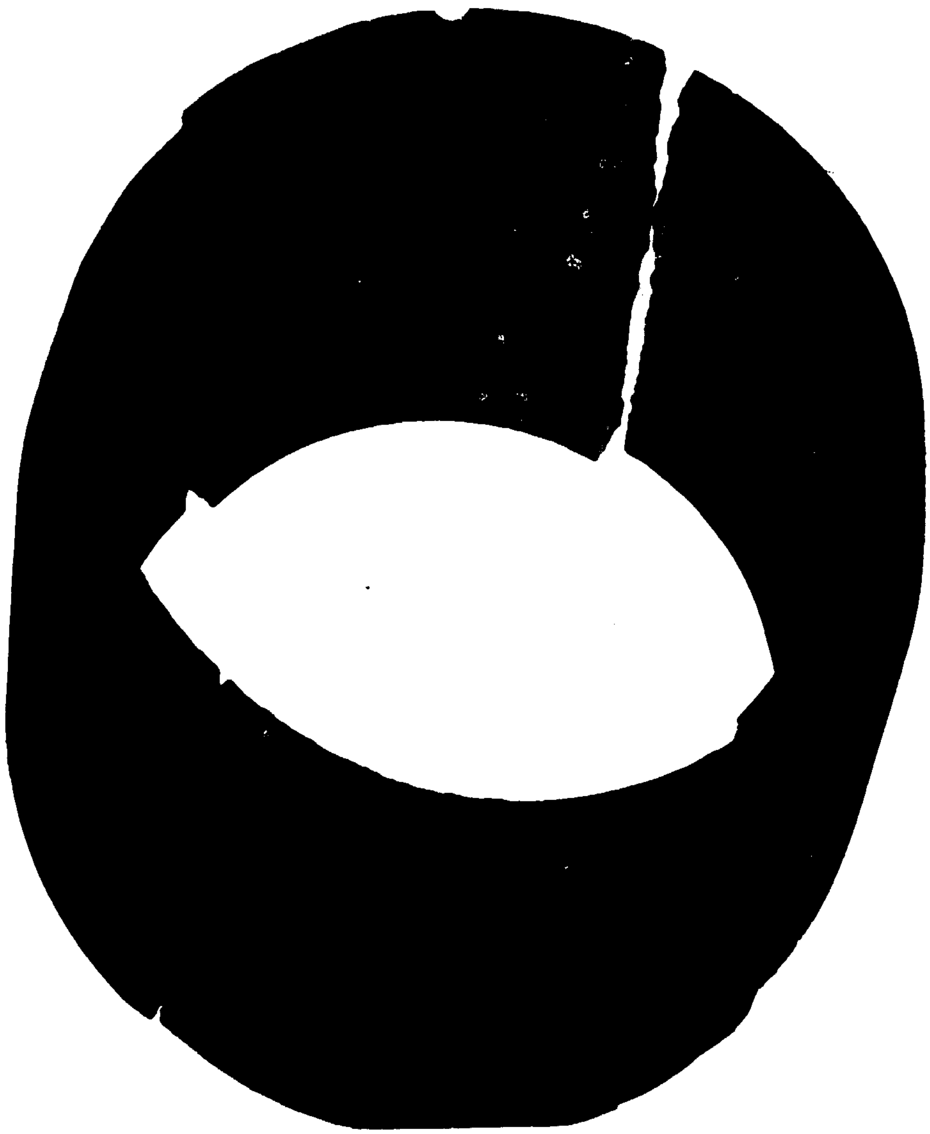


Figure 7. Post Test GA Carbon Insulation Section

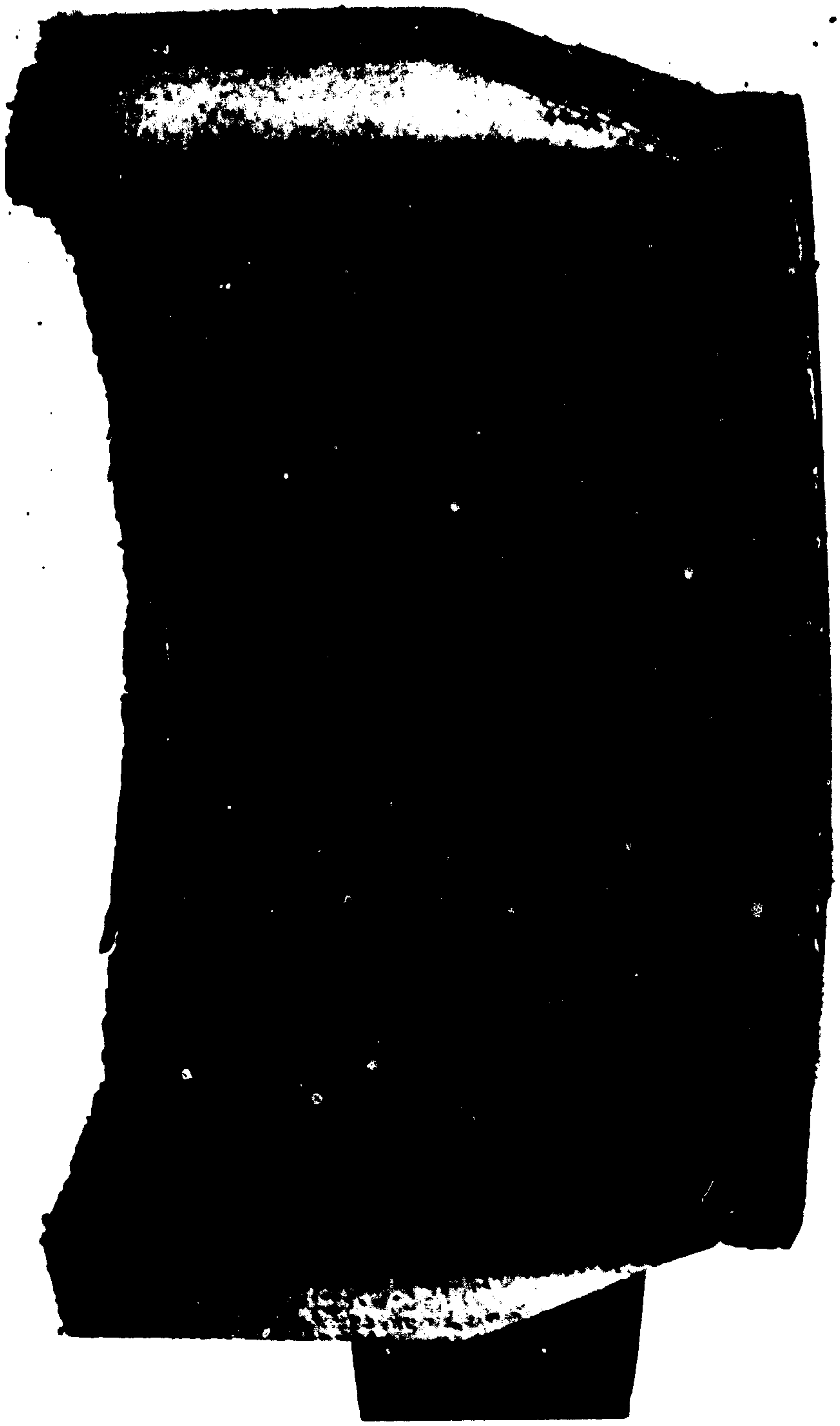
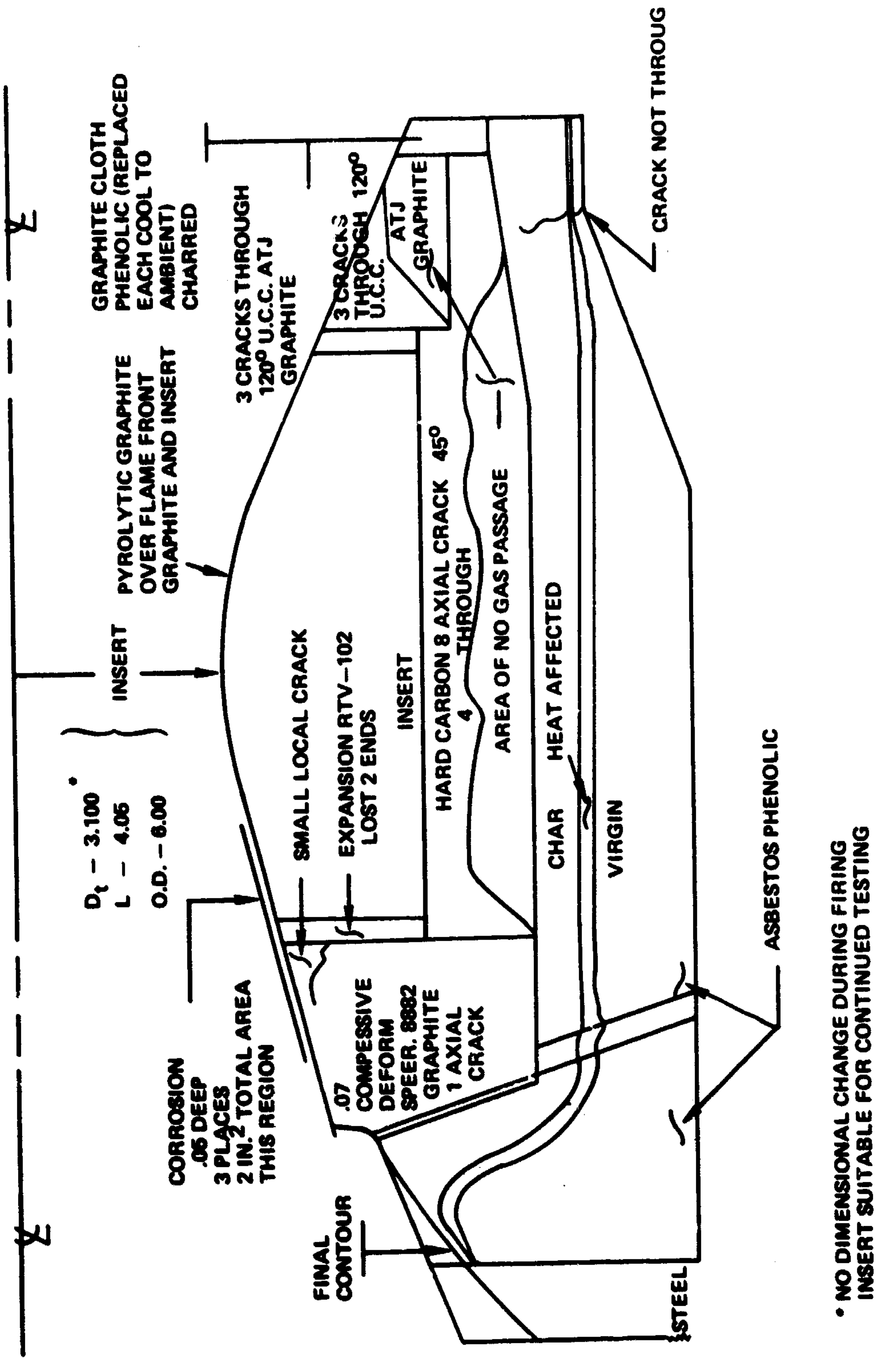


Figure 8. Asbestos Phenolic Showing Char Depth



* NO DIMENSIONAL CHANGE DURING FIRING INSERT SUITABLE FOR CONTINUED TESTING

Figure 9. Summary Diagram of Post-Test Analysis

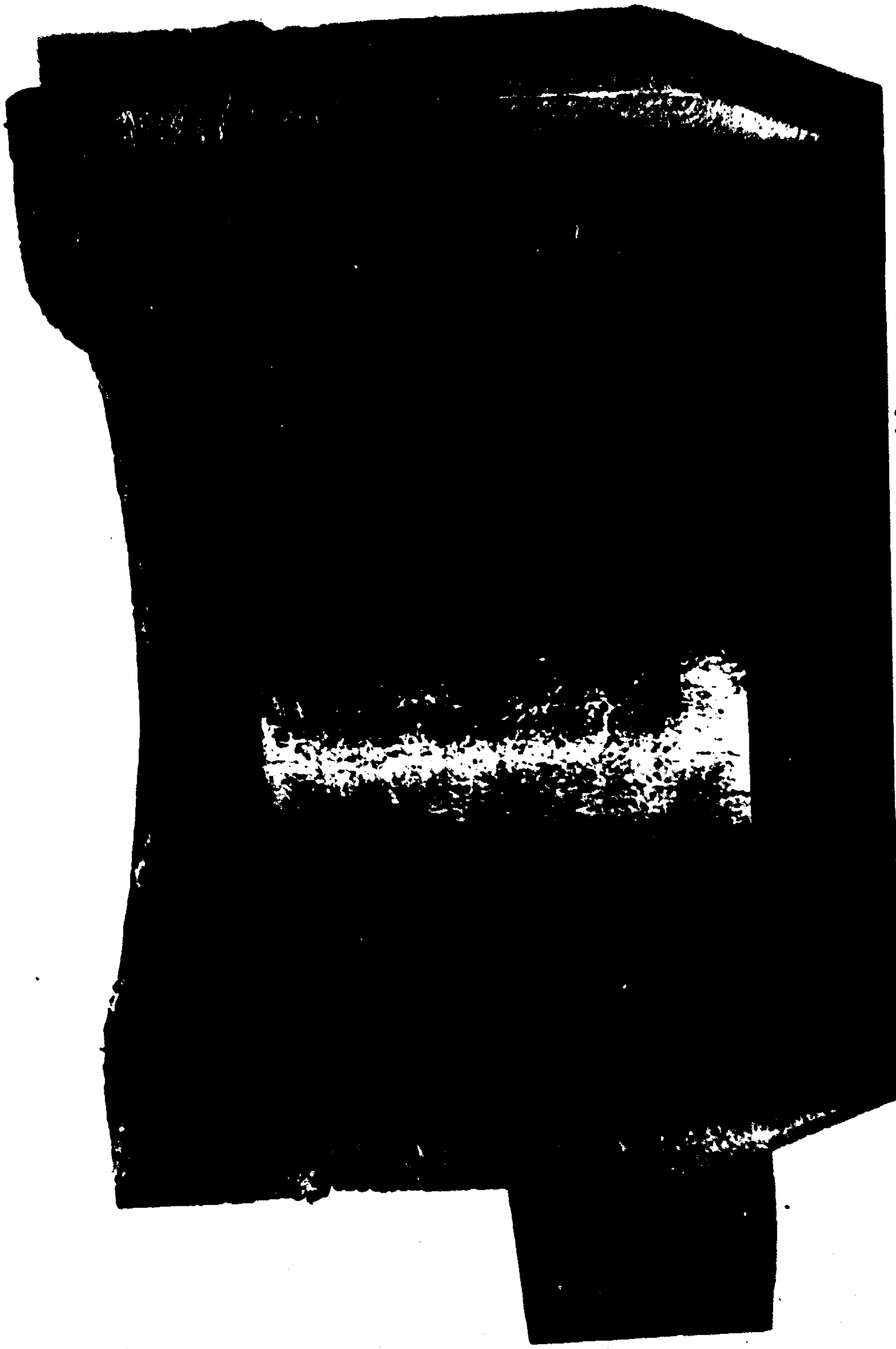


Figure 10. Post Test Cross Section of Insert

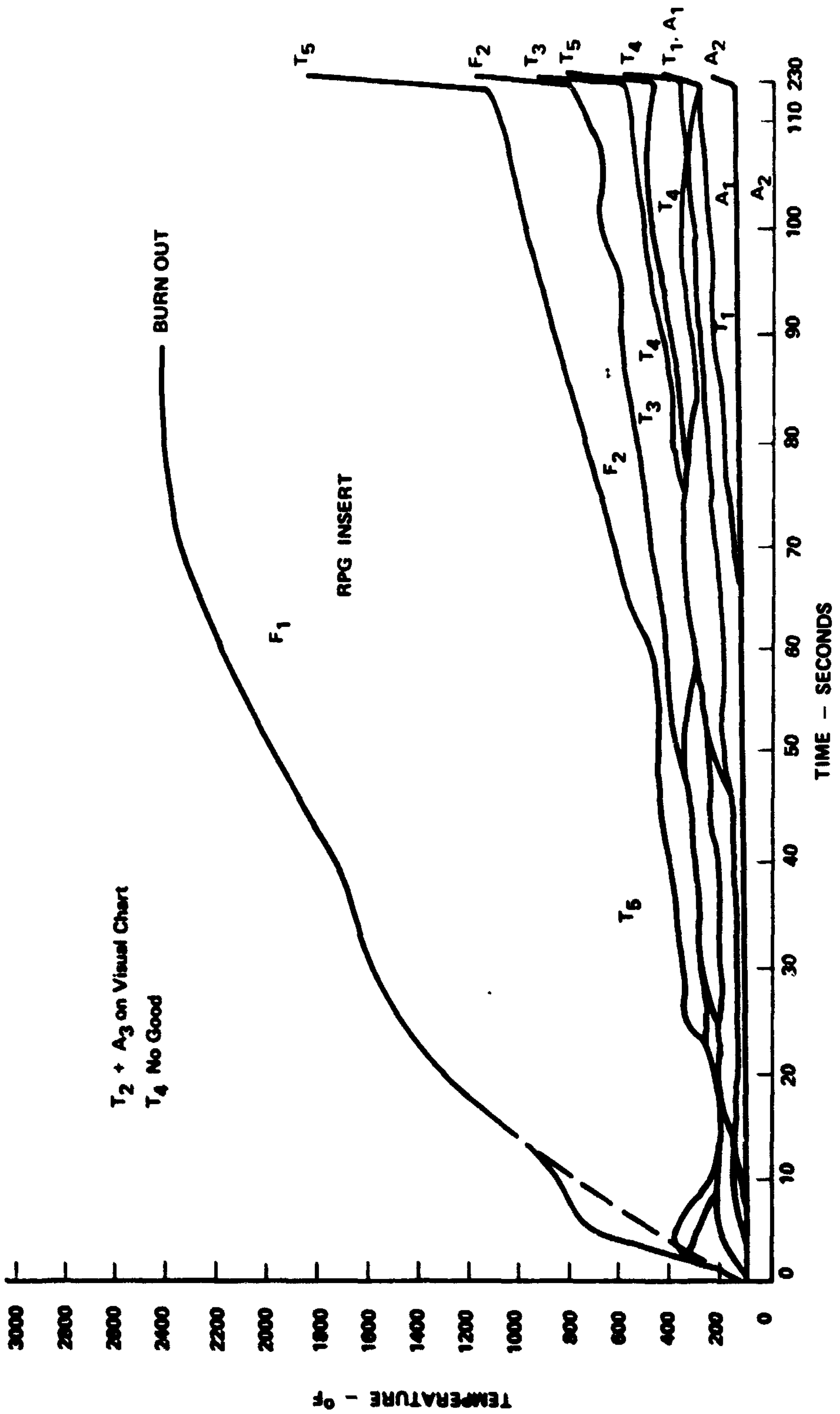


Figure 11. Thermal Response of Insert

TABLE I. THERMOCOUPLE POSITIONS

THERMOCOUPLE POSITION*	AXIAL LOCATION (in.)	DEPTH re CAN (in.) G1F3	MATERIAL CONTACTED
F-1	0.5	.75	ASBESTOS PHENOLIC
F-2	0.5	.40	ASBESTOS PHENOLIC
T-1	3.5	1.1	ASB. PHEN.-CARBON
T-2**	3.5	0.3	ASBESTOS PHENOLIC
T-3	3.5	1.1	ASB. PHEN.-CARBON
T-4	3.5	1.50	CARBON
T-5	3.5	1.65	CARBON-INSERT
T-6	3.5	1.25	CARBON
A-1	7.0	1.0	ASBESTOS PHENOLIC
A-2	7.0	0.5	ASBESTOS PHENOLIC
A-3**	8.0	1.25	ASB.-PHEN.-STEEL

*F is forward, T is throat area, A is aft; thermocouples in the same plane will be 20° apart.

**Monitor during firing.

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Project Engineer, Liquid Rocket Division. B.S. Chemical Engineering, Kansas State University, 1967. Assigned to the Air Force Rocket Propulsion Laboratory immediately upon graduation and commissioning into the Regular Air Force. Has spent his entire tour working in the Engine Research Branch, Liquid Rocket Division. Here he has been involved with the development, design, test and evaluation of rocket materials being considered for use in passively cooled engines. He has been intimately involved in several test programs evaluating materials with ClF_3, LF_2, and N_2O_4 oxidizers and amine fuels. He co-authored a paper which he presented to the 11th Liquid Propulsion Symposium, September 16-18, 1969, at Miami Beach, Florida, sponsored by the Chemical Propulsion Information Agency. He is a member of the American Institute of Chemical Engineers and the Air Force Association.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
CTF						
MMH						
Graphite						
Nozzle Insert						

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